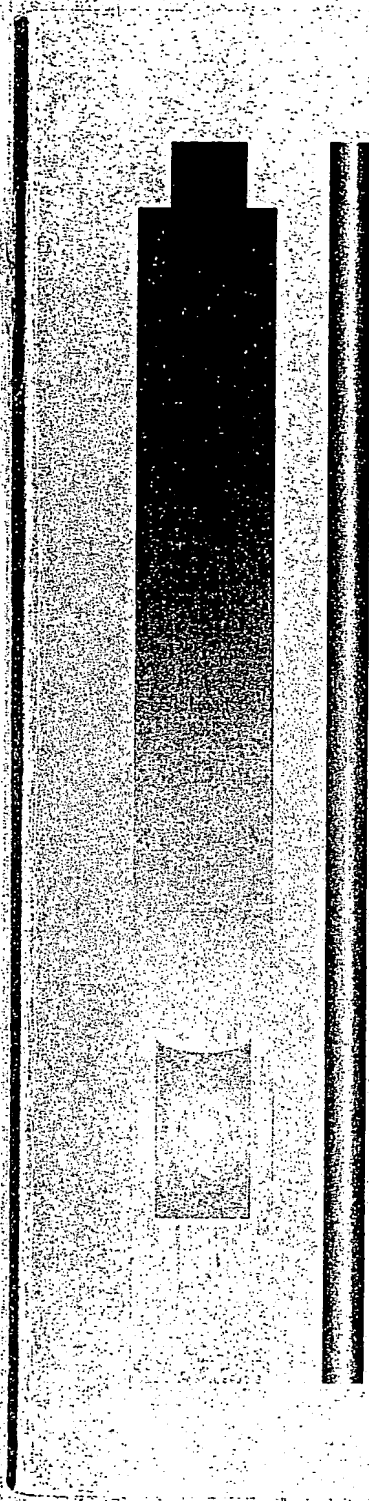




*In Volume*



# Zirconium Alloy Fuel Clad Tubing

Engineering  
Guide

Sandvik Special Metals

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# **Zirconium Alloy Fuel Clad Tubing Engineering Guide**

*First Edition  
December 1989*



**Sandvik Special Metals Corporation**

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## Acknowledgements

This Guide contains information from many sources. Those that could be referenced have been, but much of the information has been provided by members of the Sandvik Special Metals (SANDVIK) organization who have worked with zirconium products for many years. Although it is inappropriate to list all those who contributed, the following SANDVIK employees made significant contributions: Claude Stacey, Dr. Ross Bradley, Thomas Andersson, and also Craig Eucken of Teledyne Wah Chang.

A special recognition is given to John Schemel for his dedication to writing and organizing this Engineering Guide.

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The data included herein is based upon the most recent information available and, to the best of our knowledge, the use of the most accurate testing equipment and techniques. Accordingly, the use or application of this data is to be understood as a basis for recommendation, but not for guarantee.

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Sandvik Special Metals Corporation  
Kennewick, Washington USA  
December 1989

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## Introduction

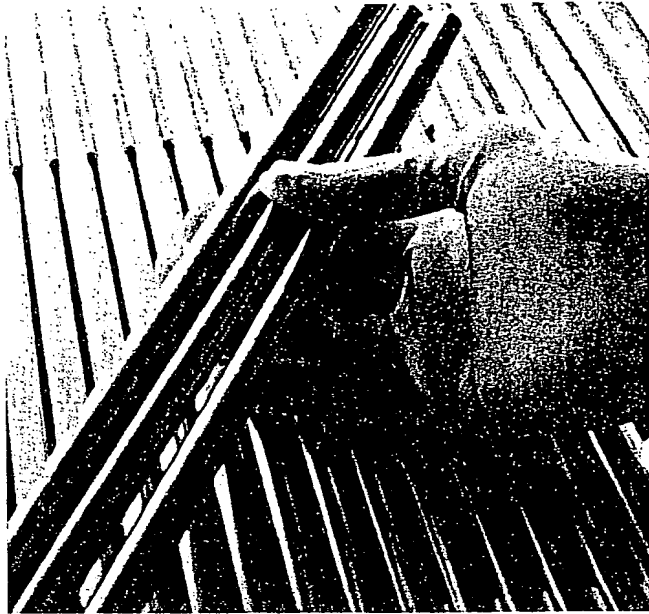
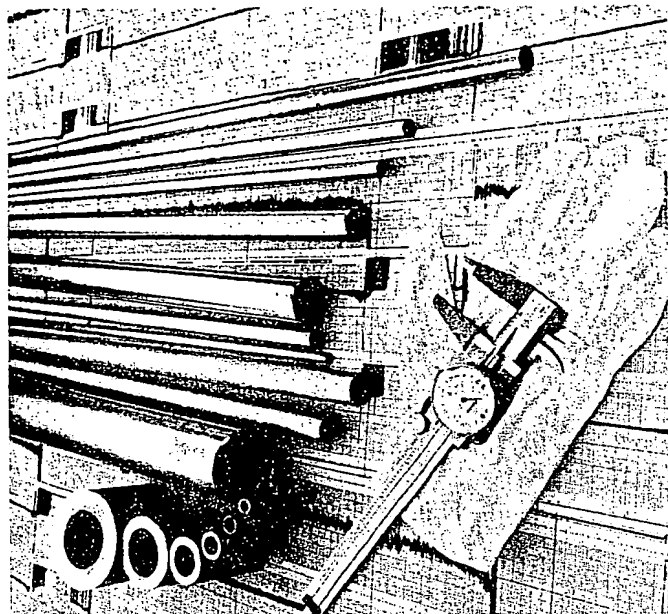
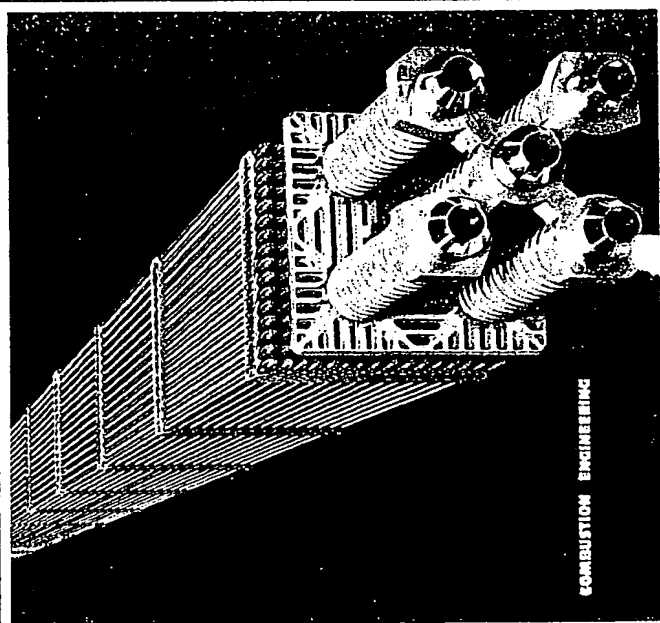
This manual has been prepared as an introduction to zircaloy fuel cladding. Its use is intended for those just becoming interested in the subject as well as a ready reference for those who routinely work with cladding. The text is general in its treatment of the subject because of the large and ever growing body of technical data that reflects the state of the art and science in 1989. Some subjects are dealt with several times so that each chapter will be reasonably complete within itself and not require constant referencing to other chapters. Each treatment will be slightly different as it comes from the perspective of the chapter. Where necessary, because of complexity or length, references to other chapters are included.

Special notation is made of processes unique to SANDVIK's way of making fuel cladding. Comparisons to conventional practice are made to assist the reader in understanding the differences and how they impact performance.

The "jargon" associated with this product is explained as each term is introduced. For instance, "zircaloy," the name was coined in the very early days at the Bettis Laboratories of the Naval Nuclear Propulsion Program and now generally represents the commercial alloys of zirconium

where tin, iron, chromium, and nickel are the major alloying elements. Zircaloy-1 was an alloy of zirconium and 2.5% tin and was never used commercially. Zircaloy-2 and -4 are in common commercial use for fuel cladding. Zircaloy-3 was a zirconium alloy containing 0.25% tin and 0.25% iron that never saw wide use, but is once again being studied. It is quite possible that the zircaloy name will continue to be extended to include all alloys with new compositions. The zirconium-1Nb alloy is often referred to as "Russian Zircaloy" since it is the standard alloy used in Russia for the same purposes as Zircaloy-2 and -4.

References listing the specific papers referred to in the text and a listing of the major sources for additional information on the subject matter covered is included in Chapter 7. The text includes specific references only where a major subject is involved to facilitate easier reading.



## Chapter 1

### *History*

While zirconium was discovered by Klaproth in 1824, our story begins in the late 1940's with the discovery that hafnium, which naturally occurs with zirconium in zircon, had caused experimenters to incorrectly calculate the thermal neutron cross section of zirconium. Researchers at Oak Ridge correctly established zirconium's cross section at 0.18 barns making it a very attractive cladding material for thermal neutron reactors.

In the early 1950's the Naval Nuclear Propulsion Program under Admiral Rickover settled on zirconium as the fuel cladding material to be used in the submarine Nautilus' water cooled reactor core. Zircaloy-2, an alloy of zirconium with tin, iron, chromium, and nickel, became the standard. It was also adopted by the engineers in Canada who were designing reactors which used natural uranium, rather than enriched uranium, for fuel. The work at Atomic Energy of Canada Ltd. (AECL) provided much of the technical data on which the commercial use of zirconium alloys was based. Unlike the Naval programs which operated under security classifications, the AECL work was widely published in open literature. Also, the Canadian work centered on tubing and commercial power reactors use tubular fuel elements.

Early U.S. power reactors used austenitic stainless steel as the fuel cladding. Chloride stress corrosion cracking of stainless steel forced the Boiling Water Reactor (BWR) engineers to shift to Zircaloy-2. Later the neutron economy of zircaloy over stainless steel made it the cladding material of choice for Pressurized Water Reactors (PWR). PWR's used a new version of zircaloy called Zircaloy-4, modified in composition to reduce the rate of absorption of hydrogen.

In Russia, an alloy of 1% niobium in zirconium became their standard for fuel cladding for both naval and commercial power reactors.

Fuel cladding for water cooled power reactors must meet a variety of technical and economic challenges. It must be corrosion resistant, have a low thermal neutron cross-section, and have satisfactory strength at the operating temperatures of the reactor core. Zircaloy met these general criteria and early fuel designs were based on the properties found in tubing produced around 1960. The push for ever more efficient fuel has been met with optimization of basic zircaloy tubing rather than a dramatic shift to another alloy system. Only the development of the Zirconium-2.5% niobium alloy for Canadian pressure tubes would lead to a new alloy; but even there, the fuel cladding remains zircaloy.

Three very significant discoveries made substantial impacts on the application of zircaloy in water-cooled reactors. The first was the discovery that nickel in Zircaloy-2 promoted absorption of hydrogen. This was very important in PWR's which use an overpressure of hydrogen in the reactor coolant which adds to hydrogen generated by the corrosion reaction. The Zircaloy-4 composition evolved with the nickel content of Zircaloy-2 being replaced with additional iron to maintain the desired corrosion resistance. The next

discovery was the effect of an intermediate "beta quench" in the manufacturing process which improved uniformity of the microstructure and reliability of the corrosion performance. Last to be discovered was the effect of metal crystal orientation on the direction in which hydrides precipitated in the tubing. Tubing made by the cold pilger or rocking process gave rise to hydride platelets that formed circumferentially around the tube leaving the tube relatively ductile. Tubing made by the drawing process commonly used for steel and other tubes produced radially oriented hydrides which made the tubing brittle. Since hydrogen was introduced into the tubing as a result of normal corrosion during service, this was an crucial effect.

There is no single set of "best" properties for a fuel clad tube. The properties must complement the requirements of the fuel design concept. The tubing manufacturer "tailor makes" the tubing to produce the properties and dimensions that provide optimum balance for each fuel design. Unfortunately, nature does not often permit one property to be set independent of the others. The attainment of a higher level in one desirable property may require some loss in another. Manufacturing parameters must therefore be adjusted to optimize the desired properties with minimum loss in other properties. For instance, it is easy to increase strength by a variety of means, but most of them drastically reduce the ductility. The means and amount of strength increase is tempered by the need to have a certain level of ductility and in no case can corrosion resistance be sacrificed. It is this complex relationship between the metal, its metallurgy, and its properties that forces a close technical relationship between fuel designers and the tube maker to get the best possible performance.

Specifications for tubing require the measurement of a number of properties essential to the design of the fuel. However, simply meeting the minimum requirements set forth in the specified properties is not sufficient to achieve optimum or, in some cases, even acceptable performance in a power reactor. In most cases, the specification for tubing includes a requirement for a process outline which defines the major process steps known to affect tube properties not easily measured by acceptance tests. The process outlines continue to evolve as new information on the process and its impact on properties and performance become known. Typically, each new tubing vendor and each major process change is subjected to an extensive qualification program to assure that the product not only meets the specification requirements, but that it also performs satisfactorily in actual service.

The sections on properties, metallurgy, manufacturing, and testing further explore the complexities of making and utilizing zirconium alloy tubing for nuclear fuel cladding.

Research and development activities are proceeding to further optimize the zircalloys as well as to develop other alloy systems. This work shows promise of even better properties to meet the challenge of more aggressive reactor environments and longer fuel residence times.

## Chapter 2

# Specifications

Nuclear fuel clad tubing is always procured to proprietary specifications peculiar to each fuel maker. These specifications generally follow the generic format of the American Society for Testing and Materials (ASTM) Standard for Zirconium Alloy Tubing, B353, or a new specification now being prepared specifically for fuel clad tubing. The fuel manufacturers insert additional requirements for their particular fuel design.

The "barrier clad" with its internal layer of unalloyed zirconium is not yet covered by an ASTM standard.

The ASTM standard includes methods for special tests used only for nuclear fuel cladding and universally accepted sections on referee testing, resolution of disputes, and many other such items. The main technical points covered by specifications are:

### 1) Composition

It is common practice for fuel designers to add several elements to the ASTM list of impurities shown in Table 2-1 to meet certain requirements based on the fuel manufacturer's past experience or the concerns of its customers. Calcium and chlorine are examples of such added impurities. It has recently come common practice to restrict



the tin content to the lower portion of the range given in ASTM specifications for Zircaloy-4 to improve corrosion resistance and to require a maximum oxygen content different from the ASTM upper limit for either alloy.

The specific effects of the alloys and main impurity elements are covered in Chapter 5 on Metallurgy.

Table 2-1

| COMPOSITION, Weight percent        |                          |                          |
|------------------------------------|--------------------------|--------------------------|
| Element                            | Zircaloy-2<br>UNS R60802 | Zircaloy-4<br>UNS R60804 |
| Tin                                | 1.20 - 1.70              | 1.20 - 1.70              |
| Iron                               | 0.07 - 0.20              | 0.18 - 0.24              |
| Chromium                           | 0.05 - 0.15              | 0.07 - 0.13              |
| Nickel                             | 0.03 - 0.08              | ---                      |
| Fe + Cr + Ni                       | 0.18 - 0.38              | ---                      |
| Fe + Cr                            | ---                      | 0.28 - 0.37              |
| Oxygen                             | 0.09 - 0.16              | 0.09 - 0.16              |
| Silicon                            | 0.005 - 0.012            | 0.005 - 0.012            |
| IMPURITIES, Weight percent maximum |                          |                          |
| Aluminum                           | 0.0075                   | 0.0075                   |
| Boron                              | 0.00005                  | 0.00005                  |
| Cadmium                            | 0.00005                  | 0.00005                  |
| Carbon                             | 0.0270                   | 0.0270                   |
| Cobalt                             | 0.0020                   | 0.0020                   |
| Copper                             | 0.0050                   | 0.0050                   |
| Hafnium                            | 0.0100                   | 0.0100                   |
| Hydrogen                           | 0.0025                   | 0.0025                   |
| Magnesium                          | 0.0020                   | 0.0020                   |
| Manganese                          | 0.0050                   | 0.0050                   |
| Molybdenum                         | 0.0050                   | 0.0050                   |
| Nickel                             | ---                      | 0.0070                   |
| Nitrogen                           | 0.0080                   | 0.0080                   |
| Tungsten                           | 0.010                    | 0.010                    |
| Titanium                           | 0.0050                   | 0.0050                   |
| Uranium (total)                    | 0.00035                  | 0.00035                  |

## 2) Mechanical Properties

Room temperature and elevated temperature tensile tests are normally required, but without any consensus as to the temperature of the elevated temperature test or to the levels of strength and ductility. Many specifications require an ambient temperature burst test with a minimum circumferential expansion required at the failure to demonstrate ductility. The most common test is a closed end test. Expansion in a burst test is larger in an open end test than in the closed end test and specified limits recognize this difference.

## 3) Corrosion Resistance

For many years a three-day test at 400C in steam was the universal acceptance test. More recently a test at about 500C to detect nodular corrosion has been added for BWR tubing.

## 4) Hydride Orientation

Specimens of tubing are required to be charged with hydrogen and examined to see if the resulting hydride platelets are oriented in the circumferential direction.

## 5) Microstructure

There is generally some sort of requirement on grain size. Direct measurement of grain size on stress relieved tubing is not possible because the grains have been elongated by the cold working. Some specifications require recrystallization of stress relieved tubing specimens in order to measure grain size.

## 6) Dimensions

Minimum and maximum values are given for the inside and outside diameters and length. Maximums are specified for ovality, bow, and squareness of cut. A minimum is specified for wall thickness.

### 7) Ultrasonic Testing

Ultrasonic testing is universally used to assure freedom from unacceptable flaws. A maximum size of a reference notch is specified.

### 8) Crystal Orientation

The hydride orientation test gives an indication of crystal orientation, but in some specifications a contractile strain ratio (CSR) test is also imposed.

### 9) Sampling Frequency

Composition, except for gases that may be absorbed during processing, is based on the analysis of the ingot from which the tubing was made. Dimensional and ultrasonic flaw testing is done in a continuous scan of each tube and other tests are generally done on two random samples from each lot of tubing.

### 10) Test Methods

Most testing is done to ASTM or American National Standards Institute (ANSI) standards or their counterparts in other countries. Cooperation between international standards organizations in specifying fuel cladding is one of the best examples of international technical exchange.

These tests and the details of their application are discussed in Chapter 4 on Testing and Inspection to assist engineers and quality assurance people in understanding the procedures and meaning of the results.

Some very important properties such as creep rate, long term corrosion behavior, irradiation growth, and resistance to iodine stress corrosion cracking are typical of those properties that can not be specified in the simple tests required on each lot of tubing. Most require test times that are too long for normal quality assurance testing. For these, reliance is placed on the process outline and its controls on processing.

ASTM has been a leader in providing a forum for consensus standards for fuel cladding not only in the U.S. but throughout the world. The subcommittees working on nuclear standards involving fuel cladding are:

- B10.02 on Zirconium and Hafnium
- G01.08 on Corrosion in Water-Cooled Reactors
- C26.02 on Fuel and Fertile Materials
- E10.02 on the Behavior and Use of Metallic Materials in Nuclear Systems

## Chapter 3

# *Manufacture*

The entire process from the ore to the tube plays a role in the performance of fuel cladding. This chapter presents background essential to understanding the metallurgical implications of the manufacturing process as well as the reasons for some of the various tests called for in the specifications. Special processing for making the barrier clad is treated in the section on extrusion and again in the discussion of beta quenching. The remaining processes are quite similar to standard cladding and the text is not complicated by mentioning all of the other considerations in making tubing from the lined extrusion.

Nearly all of the zirconium metal is extracted from zircon sand. Zircon, which is also a gem stone, is a zirconium, hafnium silicate ( $\text{Zr-Hf SiO}_4$ ) with a zirconium to hafnium ratio of 50 to 1. It occurs in beach sands all over the world, but the zircon from Australia is preferred because it contains the fewest impurities that interfere with metal production. The zircon is separated from other components in the sands by a series of standard ore dressing techniques.

## **Sponge Manufacture**

Finely ground zircon is mixed with carbon and





with any residual magnesium metal, is removed by vacuum distillation at about 1000C which produces a porous zirconium mass commonly known as "sponge." The sponge from a reduction furnace load is called a "batch." Large chunks of sponge are crushed as shown in Figure 3-3, mixed, and sampled for analysis. A group of sponge batches is selected based on the analysis and combined into a "blend" which is again mixed and sampled to determine its composition. Except for a few volatile elements like chlorine and magnesium, all of the impurities present at this stage will remain with the zirconium and end up in the zircaloy. Iron, nitrogen, oxygen, and aluminum are the most common impurities present at this stage. ASTM specification, B349, covers sponge zirconium.

Figure 3-3

Press Crushing  
Zirconium  
Sponge

Source:  
Teledyne Wah  
Chang Albany



### Ingot Manufacture

Zirconium sponge, recycle material from processing, and alloying elements are combined in various ways into an electrode for vacuum arc melting. Recycle material can be crushed, or chopped into small pieces, to be combined with the sponge and alloying elements for pressing into bri-

quettes, or it can be melted into an ingot which is then cut into slices for inclusion into the melting electrode. In both cases, extreme care is required in cleaning, sampling, and analyzing the recycle so that unwanted impurities are not introduced. Usually sponge-based briquettes and solid recycle are assembled into a long cylindrical shape and welded together as shown in Figure 3-4. Electron

Figure 3-4

Consumable  
Electrode  
Showing Sponge  
Compacts and  
Solid Recycle  
Sections

Source:  
Teledyne Wah  
Chang Albany



beam welding in vacuum or plasma arc welding in an argon atmosphere is used to prevent contamination from the air. These long cylinders are about 12 to 18 inches (300 to 450 mm) in diameter and weigh a ton or more.

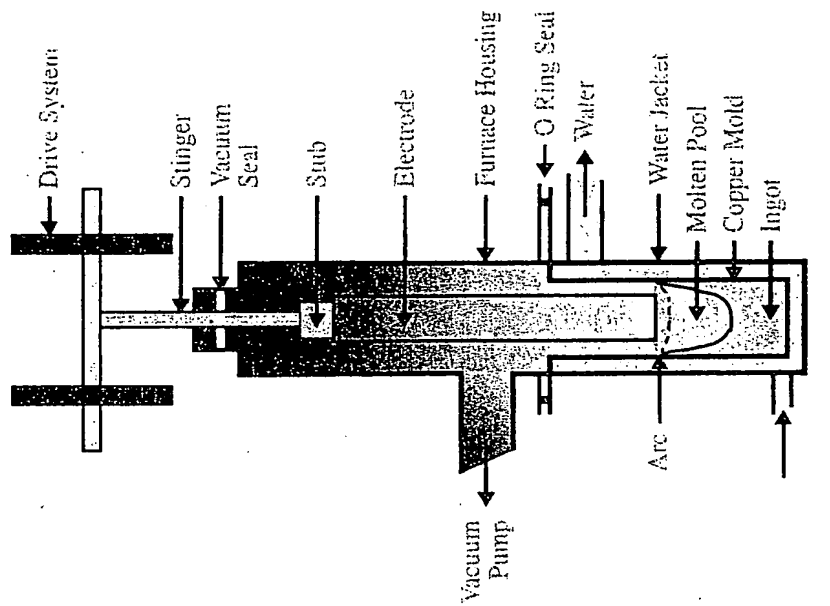
Zirconium must be melted in a vacuum or inert gas to prevent absorption of oxygen and nitrogen from the air. Consumable Electrode Vacuum Arc Melting is universally used for melting zircaloy ingots.

Zirconium, when molten, reacts with all known refractories and so it must be melted in a special way. The consumable electrode is melted into a cylindrical water cooled copper mold slightly larger than the electrode. As the arc melts the metal, a portion freezes against the cold copper mold and forms a solid layer of the zirconium alloy which acts as the container for the molten metal. The electrode is continuously consumed and the ingot builds up in the copper mold with only a portion molten at one time as shown in Figure 3-5.

Zircaloy is melted at least twice in this manner with the ingot from the first melting forming the electrode for the second melting. Much of current production is melted three times to get the best freedom from volatile elements and the most homogeneous alloy distribution. Ingots are about 30 inches (760 mm) in diameter and weigh about six tons. Since the ingot was not completely molten at one time, it is necessary to sample and analyze it at various points along its length to determine its composition. The ASTM standard calls for the first sample to be taken near the top of the ingot with additional samples taken at intervals equal to the ingot diameter down to the bottom. This recognizes that the molten pool at any time during the melting process was about one ingot diameter deep.

Figure 3-5

Pictorial  
Representation  
of Consumable  
Vacuum Arc  
Melting



Segregation is not a problem with current manufacturers as they have developed their melting techniques to produce ingots with remarkable compositional uniformity. Sampling for composition may be done under current specifications either on the ingot itself or on the material during fabrication into tubing. The sample must always be related to its position in the ingot to assure that all portions of the ingot have been analyzed. Table 3-1 shows a typical analysis report for a Zircaloy-4 ingot.

Ingots have also been tested for hardness along their length, by the Brinell method, but this practice is gradually losing significance as compositional





Figure 3-7

Rotary Forging  
Billet Stock in  
a GFM Machine

Source:  
Teledyne Wah  
Chang Albany



This is the point in the process where the "intermediate beta quench step" mentioned in Chapter 1 is performed. Either the long bar, log, or the individual billets cut from the bar are heated into the beta phase (about 1100C) and plunged into water. Billet is the name given to the part of a bar that will be put into the extrusion press. It may still be "solid" or in some instances have the hole drilled in it prior to the quench, but generally the solid billet is heated and quenched. Smaller diameter billets or billets which have a hole drilled in them have faster cooling rates when quenched. In some instances the quench baths are also modified to increase the cooling rate. A faster cooling rate has been shown to be advantageous for Zircaloy-2 used in BWR cladding, but may be detrimental for Zircaloy-4 used in PWR's.

The billet is then completely machined in preparation for extrusion into a tubular hollow, as shown in Figure 3-8. When "barrier clad" is to be produced,

Figure 3-8

Pallets of  
Machined Billets  
Ready for  
Extrusion

Source:  
Teledyne Wah  
Chang Albany



ed, the liner is inserted into the billet and welded in to place using electron beam welding in a high vacuum. Cleanliness is essential in preparing the liner and the zircaloy billet so that the bond will be complete. All through the fabrication operations, each piece is carefully identified so that its position relative to the original ingot is always known. This is a most important feature of the process as it allows structural or composition problems to be traced quickly.

Zirconium tends to gall badly when it is deformed in metal working processes. It sticks to tooling and makes for poor surface quality. One way to deal with this problem in extrusion is to encase the zircaloy billet in copper which serves as a lubricant between zircaloy and tooling. Other manufacturers have developed proprietary lubrication systems that do not use metal cladding to make extrusions. Both systems are in common use as well as combinations of them. The billet is heated to about 650C and extruded to a hollow, typically about 3.4 inches (86 mm) in diameter, as shown in Figure 3-9.



form of this process, the tube is elongated over a tapered stationary mandrel by means of two grooved rolls, also called dies, which roll back and forth in a constant cycle. It is this motion that gave rise to the term "rocking." The roll groove is tapered and the rolls are mounted in a saddle which is moved back and forth by a crank arrangement. Gears are mounted on the machine frame so that as the saddle moves, the rolls rotate in precise relation to each other and the stationary mandrel. The ingoing tube is rotated and advanced by a small increment at the beginning of each stroke of the crank. The tube diameter and wall thickness are continuously reduced in a series of these small increments. Figure 3-11a shows the arrangement of the rolls and mandrel in a typical pilger machine. Figure 3-11b shows the hollow being reduced.

Figure 3-11a

Pilger Mill Roll Stand Showing Rolls with Tapered Groove and Tapered Mandrel in Place for Tube Reduction

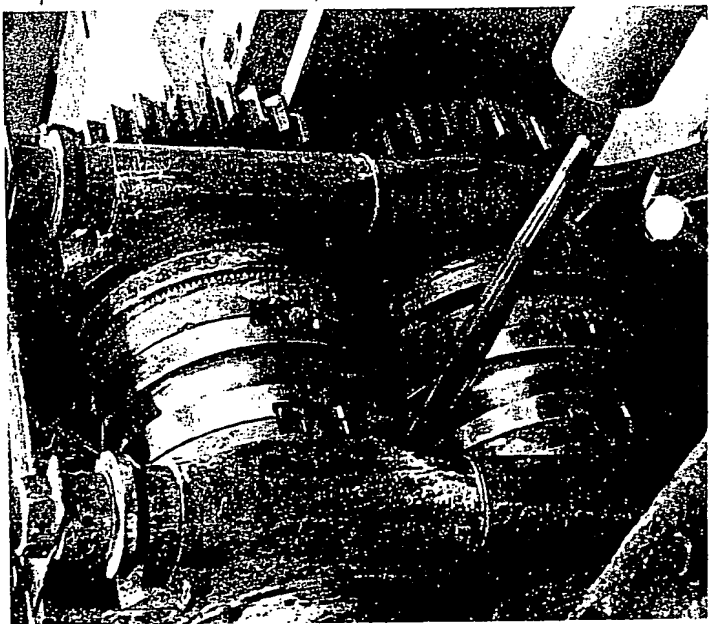


Figure 3-11b

Pilger Mill with Hollow Being Reduced by Reciprocating Rolls



Each pass through the pilger machine can accomplish up to 85% reduction in cross-sectional area, although 70% is closer to the working average. About five reduction passes are required to reduce the extrusion to fuel clad tubing size. There is a special name for a tube hollow after one cold reduction pass. It is called a TREX from Tube Reduced EXtrusion. The special name was developed because this has been a common point of sale between the zirconium metal producers and the tube makers. Tube hollow is the name for any intermediate size from the extrusion down to the hollow used for the last cold reduction pass.

After each reduction pass, the zircaloy must be cleaned and annealed to prepare it for the next cold reduction. Usually there is an inspection of the surfaces (Figure 3-12) and any small tears and cracks are carefully removed so that they will not propagate in succeeding reductions.

Figure 3-12

Video Borescope  
Used to Inspect  
the Inner  
Surface of Tubes  
and Hollows



The anneals promote recrystallization and soften the hollow. The time and temperatures of these anneals have been found to play an important role in developing optimum corrosion resistance. Zircaloy-2 and Zircaloy-4 require different heat treatments to optimize corrosion because of the differences in service environments between BWR and PWR. Zircaloy-4 is annealed at a higher temperature than Zircaloy-2 for reasons explained in Chapters 5 and 6.

The actual hollow sizes used as intermediates for each size of fuel clad tubing are quite important and will be discussed in Chapter 5 on Metallurgy. Crystal orientations depend on the ratio of wall reduction to diameter reduction, and tooling must

be carefully designed to produce the desired crystal texture. SANDVIK uses conventional tool designs for most of the reductions, but differs in the way the last reduction is made. In the patented SANDVIK process, the last reduction is made on a cylindrical mandrel that advances with the hollow. Nearly all of the reduction is in the wall thickness and not in the diameter. The combination of the advancing mandrel and the relatively large wall reduction combine to produce a strongly radial crystal texture, very uniform inside diameters, and little ovality. These features are all very desirable in the fuel clad tube.

Throughout the tube making process, tubing is kept in lots. A lot is defined in all specifications as those tubes made by the same process from a single ingot and given the final heat treatment in a single furnace load. SANDVIK goes further and keeps tube lots related to their position in the original ingot so that any composition variation will not cause variation in response to the heat treatment.

### Tube Finishing

After the last cold reduction, the tubing must be heat treated to produce the desired mechanical properties and the surfaces prepared to give the desired finish. SANDVIK pioneered a flowing nitric-hydrofluoric acid etch with a proprietary rinsing system that produces a very smooth inner surface with a minimum of residual fluoride on it as shown in Figure 3-13. As explained later, this finish provides better resistance to iodine stress corrosion cracking than other finishes, such as grit blasted or dip etched, which are also commonly applied to cladding tubes. SANDVIK also employs furnaces for the final heat treatment that are uniform within about 1 degree Celsius so that mechanical properties are extremely uniform within each tube lot and between lots. After the final heat treatment, the tubing is straightened,

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